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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

August, 1954

F. T. Howard

and

W. H Sullivan

Date Issued: SEP 8 1954

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August, 1954

The subject material of this report represents approximately half of the Laboratory's program, which is covered with some exceptions on a bimonthly schedule.

PROGRAM 2000 - SOURCE AND FISSIONABLE MATERIALS

Recovery of U^{235} - In preliminary tests with extruded Savannah River miniature slugs (5% uranium--95% aluminum, 2.4 in. long by 0.20 in. dia.) and a dissolvent 6.3 M in nitric acid and 0.005 M in mercuric nitrate, the slug surface area per inch of column height was determined. At equilibrium, after a 40-hr run in the 1-in.-dia. continuous dissolver with a 21-in.-high slug bed, the slug surface area per inch of column height was about 1.5 times greater below the first 2 in. than it was in the first 2 in., and remained about constant for the rest of the bed height. The product composition was approximately independent of the dissolvent flow rate for slug bed heights of 12 in. and greater. As the slug bed height was reduced from 21 to 12 to 6 in., the acidity of the product increased and the aluminum concentration decreased.

Experiments were carried out to determine whether the mercury catalyst in the U^{235} dissolver solution would complex the iodine present in short-cooled slugs sufficiently to prevent its escape and thus eliminate off-gas treatment. With solutions containing one-half, one, and two times the amount of iodine expected in 300-day-irradiated 90-day-cooled uranium-aluminum alloy slugs, 0.27, 0.41, and 1.28%, respectively, of the iodine was found in the off-gas. It is estimated that only 0.01% of the total iodine can be tolerated in the off-gas.

In a second trial run on the proposed first-cycle solvent extraction flowsheet, using 3/4-in.-dia. pulsed columns, a gross beta decontamination factor of 5.6×10^3 was obtained. This result represents a sixfold increase over the value obtained in the first trial run. (ORNL-1748). The increased decontamination is believed to be due to the use of highly purified solvent and to the fact that equipment performance was better. The uranium recovery was 99.97%. Studies on increasing the ruthenium decontamination indicated that ruthenium would be extracted least from a feed either 2.5 M in nitric acid or 1.2 M acid deficient. Uranium was stripped from a synthetic second-cycle extraction column effluent with 2 M ammonium citrate to give a stable product solution with a uranium concentration of approximately 240 g/liter.

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As a result of final tests on the solvent extraction flowsheet in glass 2-in.-dia. pulse columns, the column dimensions recommended for design of a plant processing 15 kg of uranium per day, with a uranium loss of less than 0.01% per column were: first-cycle extraction, 6-in. dia., 16-ft height; strip, 5-in.-dia., 17-ft height; second-cycle extraction, 2-in.-dia., 12-ft height and; strip, 2.5-in.-dia., 21-ft height.

A 15-plate 8-in.-dia. bubble cap column with a jacketed kettle reboiler was used to remove traces of tributyl phosphate solvent from a simulated strip column product and to effect a sevenfold evaporation of the aqueous stream. Mechanical operation of the equipment was satisfactory over a range of strip column product feed rates from 0.10 to 0.18 gpm. The loss of uranium was less than 0.0005% of that in the feed. (AEC Activity 2560)

PROGRAM 3000 - WEAPONS

Special Electromagnetic Separations - Relatively high enrichments of the heavier isotopes of plutonium were obtained in the fourth separation run, the results of which are tabulated below:

	Isotopic Content (atom percent)				Pu
	Pu 242	Pu 241	Pu 240	Pu 239	(g)
Feed vaporized	0.22	1.71	12.7	85.4	120
Pu 242 product	4.5	20.9	30.0	44.3	0.06
Pu 241 product	0.6	62.1	12.8	24.5	0.16
Pu 240 product	0.10	0.71	80.1	19.1	0.37
Pu 240 product	0.04	0.58	87.2	12.2	0.74
Pu 239 product	--	--	1.0	99.0	7.0

The 240 product was selectively removed from the collector in two batches. The efficiency of conversion of $PuCl_3$ vapor to well-focused Pu^+ ions was 7%. The resolution of the plutonium ion beam at the collector slots is indicated by a crossover of 2% of Pu^{239} ions to the Pu^{240} product, and a crossover of 0.6% of Pu^{239} ions to the Pu^{241} product. (AEC Activity 3601)

HOMOGENEOUS REACTOR PROJECT

Homogeneous Reactor Test (HRT) - The construction contractor for the steel-lined shielding pit began work on August 1, as scheduled. Excavation of the control room area, reactor area, and chemical separations area pits is 50% complete. The new transformer bank, which will double the present capacity, is 70% finished.

The HRT process flowsheet was revised and re-issued; no further changes are expected. The design and development of components and instrumentation is approaching 75% completion. Construction of these components (e.g., 4 sets of dump tanks, 2 condensate tanks, 2 gas separators, 2 pressurizers) can begin in September if stainless steel piping orders are delivered as expected. (AEC Activity 4109.11)

Homogeneous Reactor Experiment (HRE) - Of the zirconium, titanium, and types 347 and 304L stainless steel specimens that had been in the circulating fuel system of the HRE during most of its operation, the niobium and zirconium accounted for more than 95% of the radioactivity remaining after decontamination with several cycles of 8 M nitric acid followed by alkaline-tartrate-peroxide solution (ORNL-1703). This remaining activity could be removed only by removing the corrosion film from the metals. In the zirconium and titanium specimens the ratio of radio-niobium to radio-zirconium was about 3/2, the ratio expected from the HRE at that time. Both stainless steels had a niobium-to-zirconium ratio of 4/1, indicating that the stainless steel had selectively adsorbed niobium or that zirconium was selectively removed during decontamination.

Zirconium and titanium resisted further decontamination because they could not be descaled easily by the usual methods. The stainless steels could be descaled, and further decontaminated by a factor of greater than 100, by a 1.2 M HCl to 1.8 M H_2SO_4 reagent (ORNL-1695), by concentrated sulfuric acid in a few minutes at about 150°C, by 3% HF-20% HNO_3 in about six days at room temperature, or by 1 to 5% oxalic acid at 70°C. The latter replaced the rust-red oxide with a gray γ-Fe_2O_3 film, in 5 to 20 hr, depending on the concentration. The oxalic acid is less corrosive than the mineral acids, and it can be destroyed with hydrogen peroxide, leaving a non-corrosive waste to be stored.

HRP-Blanket Engineering Development - An aqueous slurry of ThO_2 made from thorium formate was circulated for 1450 hours in the presence of a small concentration of oxygen at 250°C. Attack on the stainless steel loop and pump was essentially limited to a small initial attack. A subsequent run with oxide supplied by the Lindsay Chemical Company showed a higher but permissible initial attack. The attack appeared to have virtually ceased when the system was arbitrarily shut down.

Concentrated ThO_2 slurries become plastic upon being pumped at 250°C and cooled to room temperature. It is not known as yet whether they are also plastic at the high temperature. A number of additives were found which reduce the yield strength of the plastic and essentially stabilize the slurry. Among these are oxalic acid, citric acid, mono-sodium phosphate, sodium hexametaphosphate and sodium silicate. (AEC Activity 4109.31)

HR Fuel and Blanket Studies - In both in-pile and out-of-pile experiments, thorium oxide catalyzed the combination of hydrogen and oxygen. When a thorium oxide slurry (806 g Th/liter) containing 4% enriched uranium oxide was irradiated (7×10^{11} n/cm^2-sec) at 210 to 220°C, the hydrogen and oxygen reached a steady-state pressure of 2000 to 2500 psi. Recombination was rapid at 250°C and above; the gas pressure dropped to essentially that of water vapor. During irradiation of a pure thorium oxide slurry for 30 days at 250°C, neither gas production nor pronounced caking was observed. In preliminary out-of-pile studies with mixed oxygen and hydrogen at a total gas pressure of 1000 psi and a mixed thorium oxide--uranium oxide slurry (500 g Th/liter) at 290 and 270°C, combination rates were approximately 0.15 and 0.05 mole of hydrogen per hour per liter of slurry, respectively. The original slurry and gas volumes were equal. A slurry of pure thorium oxide, which was prepared by pyrohydrolysis of thorium oxalate and subsequent calcination at 350°C, was 40% as efficient as the mixed oxide in combining the hydrogen and oxygen; an Ames oxide was about 10% as efficient.

Mixtures of various thorium oxide preparations and $UO_3 \cdot H_2O$ platelets (2% uranium oxide) reacted more or less completely in water at 250°C to give orange-colored solids, presumably solid solutions of UO_3 in ThO_2. Under a hydrogen atmosphere at 250°C, uranium was reduced and crystal growth was promoted.

Sodium pyrophosphate was shown to be a good deflocculant for thorium oxide prepared by calcination of thorium oxalate.

Results of large-scale experiments indicated that thorium oxide in the quantities needed for further tests and for initial operation of a homogeneous reactor may be prepared more economically and easily from thorium oxalate than from thorium formate. The oxalate was precipitated from 1 M thorium nitrate solution and calcined, first at 370°C for 5 hr and then at 650°C for 3 hr. Air-dried thorium oxalate was used in a bed 1 to 1.5 in. deep. The material shrank to about one-third its original volume during the 370°C heating, but practically no shrinkage occurred in the 650°C heating. (AEC Activity 4109.31)

HRP Miscellaneous Equipment - Tests on a carbon steel mock-up of the HRT dump-tank evaporator demonstrated that the evaporation rate would be adequate and that the evaporator would provide sufficient circulation to keep the solution in the dump tanks mixed.

Design of the HRT mock-up loop was completed, and construction of the low pressure system started.

Experiments were completed in which a 1000-gallon tank was used in simulating the steam pressure rise within the shield pit that might be caused by rupture of the reactor pressure vessel. The good agreement between calculated and observed results suggests that the theoretical basis for calculations can be applied to the full-scale HRT shield pit with considerable confidence. (AEC Activity 4109.4)

HRP Core and Blanket Systems Development Studies - Water flow tests on a full-scale HRT core model were completed. The number of screens to be used was increased over that in the original design, and the shape of the diffuser was changed to produce a stable flow pattern.

Designs for the zirconium core and stainless steel-clad reflector vessels, to be fabricated by Newport News Shipbuilding and Dry Dock Company, are essentially complete. Zirconium plate was successfully rolled, press-formed, and welded; and aluminum test model was fabricated. (AEC Activity 4109.4)

HRP Gas Handling Equipment - The HRT gas separator was fully designed, and a low pressure model was operated and proved to be satisfactory. The gas separator for the mock-up was built, and titanium is on order for two more separators for the HRT.

Slurry was run in the HRT gas separator model, which was shown to operate efficiently up to concentrations of 200 g Th/kg H_2O, the highest concentration yet tested in the separator. (AEC Activity 4109.4)

HRP Fuel Circulating Equipment - Construction of two Westinghouse model-400A canned-rotor pumps was completed. The pumps successfully passed a cold water operational test, and one of them is presently being tested on water at design conditions by the manufacturer. Loops in which the pumps can be tested at reactor conditions were designed and are being fabricated at ORNL.

Westinghouse completed the design of three pumps for blanket development and fabrication was started. The capacities of these pumps range between 200 and 300 gpm.

Helium leak testing of the 4000-gpm loop was completed. The loop system leaks less than 0.1 cc per day at $<$ 1 micron internal pressure.

A duplex, metal diaphragm-type, experimental feed pump has been under test for three weeks and appears very promising for reactor service. The pump contains a pulsating rubber member which isolates the hydraulic oil from the water which drives the metal diaphragm.

The electric pressurizer heaters for the HRT were tested and proved to be fully adequate and safe. The fuse designed to protect against overheating performed satisfactorily and the heater itself failed in an ultimate failure test, instead of the pipe wall. (AEC Activity 4109.5)

HRP Heat Removal Equipment - A contract was placed with the Foster Wheeler Co. to prepare final designs and to fabricate two 5-Mw heat exchangers for the HRT. (AEC Activity 4109.5)

HRP Instrumentation - Procurement of instruments for the HRT is well underway. Arrangements were completed for using, in the control panel, twenty-five miniature pneumatic recorders and seven controllers to be salvaged from the dismantled Orex instrumentation. A saving of approximately $6500 will result.

Detailed specifications for about 90 significant instrument components were completed. These specifications are either being circulated for project approval or have gone to the Purchasing Department.

Specifications for valves to be used in the HRT low pressure system were completed and sent to the Purchasing Department. Operators are being designed and developed for the air-operated valves because normal industrial operators contain materials which are not radiation resistant.

Detailed recommendations were prepared concerning methods of mounting ion and fission chambers about the reactor.

The basic layout of instrumentation in the control room is complete; front-of-panel layout for the main control panel is 60% complete; and preliminary lay-out of the second floor transducer and relay panel is complete. A preliminary study was made of methods of bringing pneumatic lines and thermocouple leads from the reactor into the instrument rooms. This problem is complicated somewhat by the truss-type reinforcing used in the steel-inclosed barytes and water shield.

Preliminary consideration was given to the problem of properly exhausting bleed air from the pneumatic control system and valve actuators. If exhausted into the reactor cells, this air will increase the load on the evacuation system; if exhausted into the control room some radiation hazard may exist. (AEC Activity 4109.6)

HR Fuel and Blanket Reprocessing - The equilibrium solubility of the individual rare earth fission products, when present alone in the homogeneous reactor uranyl sulfate fuel solution, was considerably higher than when a mixture of rare earths approximating that expected from fission of U^{233} was present. At 275°C the solubility of $Ce_2(SO_4)_3$ was 0.04 g/kg H_2O for the pure salt and less than 0.01 g/kg H_2O in a mixture. Under the same conditions neodymium sulfate solubilities were 0.18 and 0.045 g/kg H_2O, respectively.

The solubility of Pu(IV) in 1.0 to 1.5 m neutral uranyl sulfate was redetermined as 3.0 ± 0.5 mg/kg H_2O (the accepted value) at 250°C, and was shown to increase with increasing sulfuric acid concentration to 160 mg/kg H_2O at 0.35 m acid. The specific reaction rate constant for precipitation of PuO_2 from Pu(IV) in 1.35 m uranyl sulfate solution was 6500 $days^{-1}$ at 220°C, assuming first order kinetics, and the activation energy was 20,000 calories/mole. If the same rate is obtained under reactor conditions, the concentration of plutonium in solution in the proposed blanket will be approximately the same as the Pu(IV) solubility.

Analyses of two PuO_2 precipitates indicated that particle size in the reactor blanket will range from 0.25 to 1.5 μ. Less than 0.1 wt % of the precipitates studied was smaller than 0.25 μ and less than 10 wt % was smaller than 0.5 μ; the bulk of the material was between 0.5 and 1 μ.

In tests on a DorrClone TM-3 hydroclone separator for possible use in continuous removal of precipitated plutonium oxide from the homogeneous reactor solution, throium oxide, 90% of whose particles were less than 3.5 μ, was removed from water at 70°F with an efficiency of more than 50%. Calculations based on these results indicate that plutonium oxide particles as small as 0.5 μ may be removed from the homogeneous reactor solution at 250°C, the proposed operating temperature. (AEC Activity 4109.81)

PACKAGE REACTOR DEVELOPMENT

Army Package Power Reactor (APPR) - Invitations to bid on the 10-megawatt pressurized-water cooled reactor (Army Package Power Reactor) were sent to thirty-three prospective contractors on August 19, 1954. The specifications call for the design, construction, testing, and proven performance of the reactor under a lumped-sum type of contract. The contractors will be allowed approximately three months to answer the bid request.

The contract is being negotiated by the Chicago Operations Office of AEC. ORNL will continue to develop certain aspects of the reactor, in addition to providing consultant service to the selected contractor.

The site for the reactor was designated as Fort Belvoir, Virginia; this army post is the research and development center for the U. S. Army Corps of Engineers. (AEC Activity 4250)

AIRCRAFT NUCLEAR PROPULSION PROJECT

Aircraft Reactor Experiment - Preoperational tests of the ARE were run. Both the fuel and the sodium circuits were operated at design flow rates with water at room temperature. Operating crews were deployed on shifts, and the complete operating sequence was performed several times by each crew. Special attention was given to vacuum filling of the fuel circuit, circulating the water at design-point flow, and dumping the water in accordance with the

flush procedure to be employed with the fuel. Analogous operations were performed on the sodium circuit. These operations were useful in checking valve operation, flow meters, liquid-level probes, pressure transmitters, and other process instrumentation. The rod-cooling system was also operated, and proportioning of the cooling gas through the instrument and rod holes was effected. Dynamically, the circuits were found to function properly, and the entire system was found to be quite stable.

When all crews had become entirely familiar with the operation and all necessary information had been obtained, the circuits were drained and heat was applied to all circuits for the purpose of drying the system and checking the integrity of the electrical heater installation. The entire system was heated to a temperature of approximately 600°F. No point was allowed to exceed 600°F and some parts were heated to only 450°F. Only minor electrical difficulties were encountered and these were quickly rectified. The heating of the system proved to be much less difficult than had been anticipated and no trouble was experienced in equalizing the temperature of various parts within the desired limits.

While heat was being applied to the system, dry air (dew point -30°F) was fed into the fuel and sodium systems at the fill tanks and was exhausted through a cold trap in the off-gas system exhaust header. The purpose of the air sweeping was to remove all moisture from the system. The system was kept hot until the air at the exhaust showed a dew point of -30°F, at which time the system was considered to be dry and the heaters were turned off. This drying period required less time than had been thought necessary. The whole operation was completed in 5 days.

After the system was dry the heat exchanger housings were heated to 1100°F to check the operation of the barrier doors at elevated temperature. This test revealed that the barrier doors stuck from differential expansion and that they could have to be modified to correct this condition.

While the system was hot, it was found that kerosene (which had been used during the machining operations) was being driven out of the moderator blocks by the high temperature. Therefore a vacuum pump with a cold trap was connected to the reactor moderator volume and the kerosene was removed by vacuum distillation until all liquid that could be withdrawn by this technique had been collected in the cold trap.

As soon as the system had cooled, scheduled modifications, which had been postponed until after the water test, were begun. Preliminary testing of the system before the initial startup operations has been completed with the exception that the modified barrier doors will be tested at temperature after they are reinstalled. (AEC Activity 4401.1)

Circulating-Fuel Reactor Experiment (CFRE) - Preliminary design work on the CFRE was started with a view toward outlining the specifications for the

major pieces of equipment required. The possibilities of several dispositions of equipment and shielding are being investigated with layouts, and the characteristics of each layout are being outlined. A preliminary study of the hazards associated with each arrangement is being made as a guide in the layout work.

New plastic models of the key components of the pumps, header tank, and core were completed and are being operated with water to investigate the flow characteristics of various modifications of the basic configuration.

One of the key questions in the design of the CFRE has been the effects of severe thermal stresses in beryllium at temperatures between 1000 and 1300°F. There has been serious concern as to whether cracking or distortion in the beryllium would prove to be a major problem. A test on a 1/2 x 3 1/2 x 12 in. beryllium block was devised to investigate the effects of power (and hence thermal) cycling. The high-power-density volume-heat source coupled with transverse sodium flow through the drilled holes gave a high thermal gradient around the holes. During the course of the test the power density (and hence the thermal gradient) was cycled regularly by alternating the operating conditions from 1/3 to full power. Operation included an initial 100 hr at a constant high power density followed by power cycling at the rate of one cycle per day. The changes from one power level to the other were in all cases performed at a fairly uniform rate in a 1-min interval.

The test was concluded after 1000 hr of operation that included 36 cycles. The beryllium sample is now being examined for dimensional stability and evidence of mass transfer, corrosion, or erosion. Visual inspection of the sample after the test indicated that no distortion, cracking, or erosion of the beryllium had taken place. Calculated thermal stresses, based on the assumption of no plastic flow, gave stresses in the block of 108,000 psi for the CFRE. The thermal stresses will of course be relieved by local yielding of the highly-stressed metal. (AEC Activity 4401.1)

<u>ANP Experimental Engineering--Basic Developments</u> - Four vertical-shaft sump pumps for high-velocity, high-temperature-differential loops, used to test the corrosion of structural materials by sodium and by fluoride mixtures were fabricated. Acceptance tests were completed on three of the pumps.

The large-scale loop for isothermally (1375°F) circulating a fluoride mixture to test the corrosion and structural stability of ARE components has operated 1860 hours. An irradiated (10^7 rep from a cobalt source) V-belt was installed and has operated satisfactorily 988 hr; it is transmitting 3 hp at 800 rpm.

The critical speed range (where vibration harmonics appear) for the sheave system at the junction between the pump shaft and the impeller of the ARE-type pump was tentatively determined to be 2400 to 3000 rpm, with a maximum amplitude between 2750 and 2910 rpm. Unless tests presently underway show that there would be damage to the seals, it appears that pump shaft speeds as high as 4000 rpm should be feasible. (AEC Activity 4401.1)

ANP Experimental Engineering--In-Pile Components - The previously described horizontal-shaft sump pump for circulating fluoride mixtures in loops within the LITR or MTR has operated for more than 400 hr at 1400°F. The fluid being pumped is $NaF-ZrF_4$ (50-50 mole %). The pump delivers approximately 2.5 gpm at a head of 70 ft of fluid. This pump is being redesigned to incorporate a turbine-type impeller. In the new design, the fluid volume is greatly reduced. (AEC Activity 4401.1)

ANP Critical Experiments - A critical assembly of the reflector-moderated circulating fuel reactor, modified for simplicity in calculations by the omission of the central beryllium island, was constructed with an essentially spherical fuel core averaging 15 in. in diameter and surrounded by a beryllium reflector 13 in. thick. Alternating layers of enriched uranium metal foil and Teflon (CF_2) constitute the core and represent the mixture of fluorides in the reactor fuel. The critical loading of about 24.5 lb U^{235} is 10% higher than the mass calculated by multigroup methods. Measurements indicate that a third of the fissions are produced by neutrons having energies below the cadmium cutoff. Preparations are being made for a second assembly which will include the central beryllium island. (AEC Activity 4401.2)

ANP Radiation Damage - A new series of ten Inconel capsules is being irradiated in the MTR to compare $NaF-ZrF_4$-base fuels containing UF_3 with those containing UF_4. The 1.79 mole % UF_3 fuel generates 1100 watts/cm^3 in the A-38 position in the MTR. Two of these capsules have been examined thus far, and little evidence of corrosion has been found in the UF_3-bearing capsule. The corresponding UF_4-bearing capsule showed intergranular void formation to a depth of 1 to 2 mils. Comparison of the analytical samples of the irradiated fuels showed no significant difference in the U, Fe, Ni, and Cr compositions between the UF_3- and UF_4-bearing capsules. Since ten capsules are being irradiated, the results for these two capsules should be considered as preliminary. (AEC Activity 4401.4)

ANP-Reactor Chemistry - A revised ternary diagram with liquidus temperatures and approximate phase fields was compiled which shows the freezing pattern for all mixtures in the $NaF-ZrF_4-UF_4$ system, including those to be encountered in bringing the ARE to criticality. The liquidus temperatures for the ARE mixtures lie generally in the valley between two series of higher melting solid solutions - Na_3UF_7 in Na_3ZrF_7 and $Na_9Zr_8F_{41}$ in $Na_9U_8F_{41}$; the primary phase occurring on crystallization is always some member of the latter solid solution for mixtures of concern to the ARE.

Attempts were made to disproportionate pure UF_3 in clean helium-filled capsules of Inconel by heating for 1 hr at 1200°C followed by quenching of the specimen in air; no evidence of UF_4 or uranium metal could be found by petrographic or x-ray examination. The crystals of UF_3 were large and well formed after this treatment. Similar tests at 1300°C revealed slight evidence of UF_4 and uranium metal. At this temperature, the UF_3 had sintered but had not obviously been molten. It appears that pure UF_3 is somewhat more

stable to disproportionation than present estimates of the free energy of formation would predict.

Recent attempts to correlate conditions of preparation of UF_3-bearing melts with wet chemical analysis for UF_3 and UF_4 and results of petrographic examination show some surprising anomolies. When UF_4 dissolved in LiF, in $NaF-ZrF_4$ mixtures, or in NaF-LiF mixtures is treated under flowing hydrogen at 800°C with excess uranium metal, 90% or more of the UF_4 is reduced to UF_3. However, when this technique is applied to UF_4 in NaF-KF-LiF mixtures the reduction is only 50% complete at 800°C and, perhaps, 75% complete at 600°C. Petrographic examinations of the specimens reveal no complex compounds of tetravalent uranium; it is possible that the UF_4 is "hidden" in solid solutions or complex UF_4-UF_3 compounds in which it is not at present recognizable.

Values for the equilibrium constant for the reaction

$$Cr^{o} + 2UF_4 \rightleftharpoons 2UF_3 + CrF_2$$

in molten $NaZrF_5$ have been re-examined by using various ratios of UF_3/UF_4 with chromium metal in the charge. From the data obtained when this initial ratio is less than 3, average values of 5×10^{-4} at 600°C and 6×10^{-4} at 800°C can be computed. At ratios larger than 3 the values increase regularly; this rise is probably due to the extreme difficulty in filtration and analysis of samples containing 1 to 20 ppm of Cr^{++}. The values at low UF_3/UF_4 ratios agree quite well with the corresponding values of 4.2×10^{-4} at 600°C and 4.1×10^{-4} at 800°C obtained previously when UF_4 and chromium metal were the charge material.

Preliminary measurements of the solubility of xenon in KNO_3-NaNO_3 eutectic (66 mole % $NaNO_3$) give values of 8.5×10^{-8} and 10^{-7} moles/cm^3 at 280° and 360°C, respectively, at 1 atmosphere xenon pressure. The all-glass apparatus used was replaced with a nickel and glass combination, and measurements of xenon solubility in molten fluorides are being made. (AEC Activity 4401.5)

ANP Corrosion Research - The compatibility of the components of a Na-Be-Inconel system was studied through the use of static and dynamic tests, and it was established that these materials are compatible up to a temperature range of 1100° to 1200°F. The beryllium surface exposed to fast flowing sodium is not attacked, but in regions with stagnant sodium where the beryllium is in close contact with the Inconel, the beryllium shows attack of approximately 1 mil, which is due to alloying of the beryllium with the Inconel wall.

Corrosion tests have been initiated on materials in contact with rubidium, and preliminary tests show that Inconel suffers slight attack in contact with rubidium at 1650°F. In Inconel thermal-convection loops operated with boiling rubidium there was no mass transfer. (AEC Activity 4401.5)

ANP Metallurgy - Fabrication - A large sodium-to-air radiator with fins of high-thermal-conductivity material was constructed. The high-conductivity fins were made of copper clad with an oxidation-resistant material. The multitude of tube-to-header welds in the manifold system were made by using the semi-automatic welding machine. The tube-to-header welds were then back-brazed to give a smooth fillet joint and to remove the built-in notch at the tube-to-header weld. The back-brazing operation also ensured good closure and leak-free joints. The high-conductivity fins were brazed to the Inconel tubing with Coast Metals alloy 52. After the brazing cycle, the radiator was helium leak tested and found to be leak tight. (AEC Activity 4401.5)

ANP Metallurgy - Welding and Brazing - It was necessary to develop a brazing alloy for the fabrication of heat exchangers that was corrosion resistant to two media - the fused fluoride salt mixtures and molten sodium. One alloy that appears to be satisfactory has the composition 67% Ni, 13% Ge, 11% Cr, 6% Si, 2% Fe, and 1% Mn. It has a flow point of 2050°F. (AEC Activity 4401.5)

ANP Metallurgy - Mechanical Properties - Previous tube-burst testing of Inconel was done with 1/2-in. tubing with the wall thickness reduced to 0.010 or 0.020 inch. In recent tests 3/4 in. pipe with the wall thickness reduced to 0.060 in. was used, since data obtained in tensile-strength tests indicated that the mass of the specimen could alter the rupture life. Results obtained thus far in the tube-burst tests have confirmed the prediction that the pipe with the thicker wall would have a longer rupture life than the thin-walled tubing.

Creep tests of type-310 stainless-steel-clad copper high-thermal-conductivity fins showed that this material will withstand a stress between 500 and 1000 psi for 1000 hr without loss in properties due to cracking and oxidation of the copper cores.

An 80% Ni-20% Mo alloy, which is being studied as a possible substitute for Inconel in circulating-fuel reactors, was stressed at 5000 psi in argon at 1500°F. It ruptured in 760 hr with 5% total elongation. The following table shows a comparison with Inconel and type-316 stainless steel.

	Stress (psi)	Rupture Time (hr)	Elongation (%)
Inconel	5000	540	29
Type-316 Stainless Steel	5300	2900	8
80% Ni-20% Mo	5000	763	5

It is reported in the literature that, at this temperature and stress, Hastelloy B (28% Mo, 65% Ni, 7% Fe) tested in air would have a rupture life in excess of 10,000 hr. (AEC Activity 4401.5)

ANP Heat Transfer Research - Additional heat transfer measurements on forced-convection molten NaF-KF-LiF (11.5-42.0-46.5 mole %) were made. Measurement of the thermal conductivities and thickness of insoluble deposits on the inner walls of Inconel heat transfer tubes made it possible to calculate the thermal resistance of the deposit (K_3CrF_6). This calculated value is in good agreement with values previously deduced from the heat transfer measurements. A hydrodynamic flow system to be used for studying the reflector-moderated reactor flow structure was tested. A mathematical study was made of the temperature structure in converging and diverging channel systems whose fluids have volume heat sources. (AEC Activity 4401.5)

ANP Physical Properties Research - The enthalpies and heat capacities of $NaF-ZrF_4-UF_4$ (65-15-20 mole %) were determined; the heat capacity in the solid state over the temperature range 90° to 614°C was found to be 0.17 cal/g.°C and the heat capacity in the liquid state over the temperature range 653° to 924°C was found to be 0.20 cal/g.°C. The enthalpies and heat capacities of $LiF-NaF-UF_4$ (57.6-38.4-4.0 mole %) were also obtained; the heat capacity in the solid state over the temperature range 97° to 594°C was found to be 0.23 cal/g.°C, and in the liquid state over the range 655° to 916°C, it was found to be 0.53 cal/g.°C. The thermal conductivities of three fluoride mixtures in the solid state at normal temperatures were determined. The conductivity of $NaF-KF-UF_4$ (46.5-26.0-27.5 mole %) was 0.7 Btu/hr·ft^2 (°F/ft), that for KF-LiF-NaF UF_4 (43.5-44.5-10.9-1.1 mole %) was 2.0 Btu/hr·ft^2 (°F/ft), and that for $LiF-KF-UF_4$ (48.0-48.0-4.0 mole %) was 1.4 Btu/hr·ft^2 (°F/ft). (AEC Activity 4401.5)

ANP Analytical Studies - A successful potentiometric titration of UF_4 in molten $NaZrF_5$ with powdered metallic zirconium was accomplished by means of polarized platinum electrodes. The observed end point occurred at a weight of zirconium which corresponds to one-half an equivalent of zirconium metal per mole of UF_4.

Polarographic studies of uranium fluorides in $NaZrF_5$ and NaF-KF-LiF-base fuels, which were dissolved in molten ammonium formate at 125°C, revealed that U(IV) was not reduced at the dropping-mercury electrode. The only reduction wave that was obtained was identified as that for the reduction of U(VI). Ammonium formate is unique in that it is an excellent solvent for fluoride salts. At least 500 mg of a $NaF-KF-LiF-UF_3-UF_4$ mixture is soluble in 100 ml of molten ammonium formate. Uranium trifluoride that has not been complexed with alkali-metal fluorides is essentially insoluble in the molten formate.

It was shown that UF_3 is converted quantitatively to UF_4 and UO_2 in 4 hr at 300°C by oxygen according to the reaction

$$4UF_3 + O_2 \longrightarrow 3UF_4 + UO_2.$$

Under the same conditions, UF_4 was partially converted to an oxide of uranium

totaling about 11% of the sample. This oxide formation precludes any possible determination of UF_3 by this method.

Calibration measurements were completed on the apparatus for the determination of oxygen as metallic oxides in reactor fuels. A curve was established over the range 0.03 to 0.25% oxygen. (AEC Activity 4401.5)

ANP Shielding Analysis - Application of the Monte Carlo method to the calculation of gamma-ray penetration of crew shield sides was worked out and the method appears to be quite satisfactory for this type of problem. Considerable progress was made in understanding measurements of ground and air scattering obtained with the Tower Shielding Facility (TSF). Expressions were derived which describe the effect of ground interference with the air-scattered flux, and thus it is now possible to estimate ground-scattered radiation both at the TSF and in an airplane at landing and takeoff. Calculations were set up for evaluating multiply-scattered radiation in air. The values obtained will be of considerable importance in studies of the highly assymetric but light shields of several current designs. (AEC Activity 4401.7)

ANP Shielding - Lid Tank Shielding Facility (LTSF) - Measurements in a continuous carbon medium, obtained by dissolving sugar ($C_{12}H_{22}O_{11}$) in the water of the Lid Tank, have resulted in a value of 0.750 barn/atom for the removal cross section of carbon. This is to be compared with the value of 0.81 ± 0.05 obtained from measurements behind solid graphite placed against the source plate. (AEC Activity 4401.7)

ANP Shielding - Bulk Shielding Facility (BST) - Carbon removal cross section values obtained from measurements made behind various thicknesses of graphite against the BSR are 0.82 barn for a 1-ft slab, 0.84 barn for a 2-ft slab, and 0.80 barn for a 3-ft slab. These values are in good agreement with the LTSF values, above. (AEC Activity 4401.7)

ANP Shielding - Tower Shielding Facility (TSF) - It was previously estimated (ORNL-1748) that the contributions of ground-scattered neutrons to the total air-scattered radiation measured at 200 ft is not greater than 3%. This estimate was based on the assumption that all the neutrons were singly scattered in air. Further analysis now indicates that multiple scattering is too important to ignore, and the ground-scattered contribution may be as high as 5%.

Tests with the reactor shield section of the GE-ANP R-1 divided-shield mockup and the TSF detector tank were started at the TSF. Measurements as a function of the reactor-detector altitude indicate that the ground-scattered radiation at the maximum altitude is considerably higher than the 2 to 5% indicated by the differential experiments (see above). Some increase was to be expected since the shielding beneath the reactor in the mockup is much less than that in the reactor tank. In later measurements the TSF detector tank will be replaced by the GE crew-shield mockup.

A technique which minimizes manpower and time requirements was developed for obtaining and publishing much of the experimental data from the TSF. The data are automatically plotted on a 30-in. square Variplotter and then are prepared in report form by a photographic method which requires no drafting or tables of data. Multiple copies can be obtained as a record of the data and for any further processing. (AEC Activity 4401.7)

GENERAL REACTOR RESEARCH

General Reactor Radiation Damage - The disorder and lattice expansion in single crystals of beryl ($Be_3Al_2Si_6O_{18}$) were observed after successive bombardments totalling 2.8×10^{20} fast neutrons/cm^2. Some long range order persists after the maximum irradiation, and it is seen that the crystal structure is considerably more stable under neutron irradiation than structurally similar quartz. However, the anisotropy of the lattice expansion is common to both.

Piezoelectric tourmaline, another quartz-like structure, is rendered amorphous by an irradiation totalling 7.8×10^{19} fast neutrons/cm^2. The disordering process is probably due to two separate mechanisms. B^{10} nuclei, occupying normal crystal lattice sites and having a large cross section for thermal neutrons, undergo an (n,α) reaction involving the ejection of an energetic α particle with a range of $\sim 30\ \mu$ in this crystal. Displacements are produced by this α particle and its recoil knock-on, as well as by direct fast neutrons and their knock-ons. It is believed that the latter effect is more predominant since the (n,α) reaction is restricted to thin surface layers. (AEC Activity 4540)

Dissolution Studies - In tests in the 5.5-in.-dia. continuous dissolver, packed heights of 4, 6, and 10 ft were shown to give dissolution rates of 5, 10, and 22 kg of uranium per day, respectively. The dissolvent was 6.5 M nitric acid that was 0.005 M in mercuric nitrate; the slugs were extruded 5% uranium--95% aluminum alloy Savannah River type, 12 in. long by 1-1/8 in. dia. In a test dissolution of pure aluminum bar stock, made to determine some of the data needed for design of a trickle-type dissolver, the mercury concentration of the reflux stream was shown to be equal to or greater than that of the feed stream. The dissolution rate decreased about 15% when the mercury concentration was decreased from 0.005 to 0.0031 M. The rate of slug addition to maintain a constant packed height appeared to be constant while steady-state conditions were being approached. When a run was started with fresh slugs and the feed rate was constant, the product concentration increased considerably during the first 1 or 2 hr, and then increased gradually.

When caustic solutions of irradiated slugs were batch-centrifuged to separate the suspended uranium oxide, the bulk of the fission products followed the solids. Gross beta and gamma decontamination factors of 10-25 and 5, respectively, were obtained for the waste supernatant. These factors are about the same as those obtained when fission-products were added to caustic solutions of unirradiated uranium (ORNL-1748). Cesium, iodine, and ruthenium were the

major radioactive contaminants remaining in the waste. In attempts to decontaminate the waste further, prior to storage, the aluminum was precipitated as $Al_2O_3 \cdot 3H_2O$ by treating the solution with carbon dioxide. The ratios of beta and gamma activities in the supernatant to those in the precipitate were 6.3 and 2.9, respectively; 70% of the zirconium and 90% of the niobium in the waste followed the precipitate.

From synthetic solutions of the type expected in nitric acid dissolution of stainless steel--clad uranium fuel elements, from which the carburized cladding had been removed with copper sulfate solution, the uranium was extracted with 6% tributyl phosphate. The uranium loss was less than 0.01%, and the gross beta and gamma decontamination factors were 10^5 and 3×10^4, respectively. Five extraction and seven scrub stages were used. The feed, which was 2.5 M in copper nitrate and 0.07 M in ferrous nitrate, contained 13 g of uranium per liter. The scrub was 2 M in nitric acid, 0.02 M in sulfuric acid, and 0.4 M in urea. The ferrous ion was used in the feed to increase the ruthenium decontamination of the product, and the urea in the scrub to decrease the amount of NO and NO_2. (AEC Activity 4581)

PROGRAM 5000 - PHYSICAL RESEARCH

ISOTOPE PRODUCTION

Stable Isotope Production - Collection of an estimated 430 grams of chromium isotopes (masses 50, 52, 53, 54) was completed in July. Through the use of a new type, large two-arc ion source, the collection rate for chromium isotopes was increased by a factor of three over the collection rate achieved with the single-arc Beta-type ion sources used in previous chromium separations. The processing of calcium isotopes (masses 40, 42, 43, 44, 46, 48) was started in August, again using the larger ion source as a means of increasing the production rate. Isotopes of chromium and calcium will be used as cyclotron targets in the preparation of much needed radioactive isotopes which are not otherwise readily available.

With the completion of chemical refinement of Gd 152, Gd 154, and Gd 155, all isotopes of gadolinium are available for the first time. All of these are better than 99.8% Gd_2O_3 in chemical purity. Newly separated lots of Cu 65, Zr 90, Zr 91, Zr 92, Zr 94 and Sm 144 were also added to the inventory. Europium and dysprosium are being extracted from mixtures of rare earths by the use of newly installed facilities in Building 9731.

Small quantities of pure Ho_2O_3, Sc_2O_3, RbCl, and CsCl were prepared for spectrographic standards from crude commercial materials.

Twenty-one isotope samples were spectrochemically analyzed for a total of 481 impurity determinations.

Sixty-two shipments were made consisting of 49 different isotopes of 28 elements. (AEC Activity 5121)

Scintillation Spectrometry - The total absorption spectrometer which employs an anti-coincidence tank surrounding a central detector was improved and is now in regular use. A NaI crystal, equivalent to a sphere of NaI 5-1/4 inches in diameter, is now being used as the central detector. With this arrangement the spectrum produced by a Zn^{65} source has 88% of all pulses in the "photo" peak, and no point in the Compton distribution has an intensity higher than 1/50 of the intensity in the peak. With this good discrimination against the Compton distribution, the combined internal and external Bremsstrahlung spectrum of Y^{91} was obtained, and no correction was necessary for the Compton distribution of the weak 1.2-Mev nuclear gamma ray.

Investigations are in progress on a sample of K^{40} which was obtained by the combined efforts of the Canadian and U. S. Atomic Energy Commissions. The results are being analyzed, and the ratio of beta-to-gamma emission will be obtained. There appears to be evidence for a very low intensity gamma ray in the vicinity of 800 kev. Coincidence measurements on this sample at 180° and 90°, to search for actual positron emission, show a peak of annihilation radiation so weak that it is uncertain whether or not all of it is contributed by pair production in the source.

Bi^{207} was investigated with the scintillation spectrometer and the 1.46-Mev gamma ray reported by Prescott, Proc. Phys. Soc. A67, 540 (1954) was not found. In addition, a peak at 2.4-Mev, which was reported by the same author, was found at 2.34 Mev but may be attributed to pulses of the 1.78 and 0.570-Mev gamma rays occurring coincidently in the same detector. A limit of less than 0.5% of the intensity of the 1.78-Mev gamma ray may be placed on the intensity of a gamma ray at 2.34 Mev. Quantitative coincidence measurements show no prompt 1.067-Mev K-X-ray coincidences, which are not accounted for by the conversion of the 0.570-Mev gamma ray. In addition, no prompt 1.78-Mev K-X-ray coincidences were found which are not similarly accounted for, indicating, possibly, only L-capture to the state at 2.38-Mev above the ground state.

Studies are being made of solution phosphors to improve the performance of the anti-coincidence tank counter. More dense phosphors, and preferably those containing higher atomic numbers, would improve the performance by giving greater gamma ray absorption with less total weight of the tank. It was found that tri-m-tolyl phosphate makes an intrinsic solution phosphor medium, and with the addition of p-terphenyl and α-naphthylphenyloxazole, there is about a 50% increase in density and pulses are obtained which are half as large as those from the standard solution phosphor. A few percent of $HgCl_2$ can be added with the aid of a small amount of alcohol, and this addition considerably increases the photoelectric capture. (AEC Activity 5200)

High Voltage Program - The background from (n,p) reactions in the neutron spectrometer was reduced by placing platinum apertures at the radiator position. In this spectrometer, neutrons knock out protons from a polythene radiator. The protons pass through two proportional counters and are stopped in a NaI crystal. The two proportional counters are constructed so that only one entrance and one exit define the proton beam. Triple coincidences from the three counters gate a multichannel analyzer which "looks" at the proton spectrum in the NaI crystal. This spectrometer analyzes 14-Mev neutrons with 4% energy resolution and gives an absolute measure of neutron flux.

The spectrometer was used to observe the neutron spectrum from the $Li^7(d,n)Be^8$ reaction at 120 deg and the absolute differential cross section for this reaction, which leaves the Be^8 nucleus in its ground state and first excited state. At 120° the neutron spectrum indicates only one excited state up to 9-Mev excitation. This is in agreement with data previously obtained at 0° with this spectrometer, but is in disagreement with published spectra showing several excited states. For 2-Mev deuterons the differential cross section for the ground-state group of neutrons decreases from 3.3 ± 0.3 mb/steradian at 0° to approximately 1.7 mb/steradian for angles greater than 50°. The cross section for the first excited state decreases from 7.0 ± 1.0 mb at 0° to approximately 5 mb/steradian for angles greater than 80°. In addition the spectra indicate a large neutron continuum from the three-body breakup of Be^9. The forward peak in the differential cross section is consistent with Butler stripping curves for capture of p-wave protons.

The gamma rays from the proton bombardment of C^{13} were observed with a large 3" x 3" NaI crystal having a resolution for the Cs^{137} photo peak of 8%. The capture γ-rays exhibit a resonance at an incident proton energy of 3.11 Mev, 30 kev width. The angular distribution of the ground-state transition (10.43 Mev) was measured; it is best fitted by the expansion $1 - A_2P_2(\cos\theta)$ where $A_2 = 0.40 \pm 0.02$. This result is consistent with the capture of d-wave protons forming an excited state of $J = 2^-$, and emission of electric dipole radiation to the ground state of N^{14} (1^+). In the channel spin notation, the mixing ratio is 3 to 2 for channel spin 0 to channel spin 1; however, both j - j coupling and Russell-Saunders coupling predict the proper distribution. (AEC Activity 5200)

86-Inch Cyclotron, Nuclear Physics - Angular distributions of neutrons from 22-Mev proton-induced (p,n) reactions were measured for ten target elements by using silver and aluminum threshold detectors. The results indicate that a large fraction of (p,n) reactions go by direct, rather than compound-nucleus, interaction.

Measurements of fission-fragment angular distributions from U^{233} fission were completed, and measurements were begun on Th^{230}. Measurements of the fission cross section for Th^{230} and U^{233} were made.

Projects on measurements of cross sections for (p,n), (p,α), (p, He_3), and (p,2p) reactions were undertaken or continued. The project on angular distributions of alphas from (p,α) reactions was terminated without publication. The conclusion was that the angular distributions are essentially symmetric about 90°, but that there is probably an excess at small angles. (AEC Activity 5200)

86-Inch Cyclotron, Applied Physics - Two new cyclotron targets were designed and constructed, a grazing-incidence type for irradiating metals of medium and high melting points and a water-cooled capsule target for non-metallic substances. The thin walls of the aluminum capsule reduce the proton energy by only 2 Mev. Both targets were designed to facilitate the handling and chemical processing of radioactive materials.

Tests are underway to determine the adaptability of a fixed frequency cyclotron for the acceleration of various ions. Normally a cyclotron is tuned for the first mode, that is, during each half-cycle of applied radio frequency the protons transit 180 degrees of their orbit. Ions requiring 3, 5, 7, etc., half cycles rf to transit 180 deg can also be accelerated in a two-dee cyclotron; the cyclotron is tuned by adjusting the magnetic field. In tests with the 86-inch cyclotron both N^{3+} and N^{5+} were accelerated at the same resonance conditions. As much as 30 μa of N^{3+} was accelerated at the fifth mode to 10-12 Mev. At the same time, a small output ($\sim 10^{-3}$ μa) of N^{5+}, accelerated to $>$ 30 Mev at the third mode, was detected. The energy and identity of these higher energy ions was determined by the activation of beryllium foils through absorbers and by activities (F^{18} and Na^{24}) produced in carbon.

Routine radioisotope production bombardments included: five hours on lithium metal, yielding 520 mc of Be^7; twenty-five hours on chromium producing $\sim$15 mc of Mn^{54}; and 8 mc of Sr^{85} produced by irradiating RbCl in the capsule target for four hours. Gallium 67 production for the ORINS Medical Division continued with a bombardment every third week, each producing $\sim$350 mc. As a part of the UT-AEC Agriculture Research Program, fluorine 18 (1.87 hour half life) which is produced via the (p,2n) and (p,n) reactions on F^{18}, is being used in a study of fluorine uptake and distribution in sheep. A one-hour bombardment of NaF in a capsule produces roughly 300 mc of F^{18} and 10 μc of Na^{22}. For the above application, the Na^{22} causes no difficulty. (AEC Activity 5200)

86-Inch Cyclotron, Operation - Major details of the system for deflecting the proton beam of the cyclotron have been engineered and the shop work is well underway. The system includes both an electrostatic deflector with water-cooled septum and an electromagnetic "escape" channel. The cyclotron shut-down for deflector installation is scheduled to begin October 8 and is expected to continue for about four months. (AEC Activity 5200)

63-Inch Cyclotron, Physics - The equilibrium charge distribution for nitrogen ions from 6 to 26 Mev was measured in thin plastic foils, approximately 20 $\mu g/cm^2$ thick. Only the three most tightly-bound electrons were captured and lost over this energy range. Over the energy range investigated the ratio of the loss to capture cross section for the K electrons was dependent on the ion velocity and was found to be proportional to $V^{3.4}$, while for the most tightly-bound L electron it is proportional to V^2.

Thin non-equilibrium foils, $\sim 1\ \mu g/cm^2$ thick, were used to measure the cross sections for capture and loss of electrons at 26 Mev. The values are given below:

Capture of sixth electron	=	$2.4 \times 10^{-17}\ cm^2$
Loss of sixth electron	=	$8.4 \times 10^{-18}\ cm^2$
Capture of seventh electron	=	$1.4 \times 10^{-17}\ cm^2$
Loss of seventh electron	=	$1.1 \times 10^{-17}\ cm^2$

The absolute values of the cross sections are known only to within a factor or two, due to the difficulty in measuring foil thicknesses of the order of 1 microgram/cm^2. (AEC Activity 5200)

Spectroscopy Research Laboratory - Improved sensitivity of microwave equipment permitted observations of the high $7_{16} \rightarrow 7_{17}$ transition of vinyl fluoride at 42,440 megacycles and the detection of transitions in the 0.5%-natural-abundance compound $C^{12}H_2C^{13}HF$. Design of a temperature-controlled absorption cell for the infra-red analysis of low concentrations of H or of D in water was completed. Evaluation of several Landé g factors in the Zeeman spectrum of plutonium revealed significantly wider departures from LS coupling than in samarium, its first rare-earth analogue. Evaluation of Fabry-Perot interferograms of the red line from separated isotopes of cadmium show, for successive even-mass isotopes, spectrum shifts of the order of one ten-millionth of the wavelength. The measurements do not agree with data of Russian scientists, but are more complete and show anomalous nuclear volume shifts which appear to correlate with nuclear shell closings. Investigations of complex element matrices (rare earths and steels) by x-ray fluorescence analysis techniques demonstrated conclusively the seriousness of impurity effects in the interpretation of the data. A spark source was developed for excitation of Th(III) in a magnetic field for the study of important infra-red combination lines. (AEC Activity 5200)

Mass Spectrometer Laboratory - A standard type gas discharge tube was attached to the vacuum system of the General Electric mass spectrometer, and cataphoresis studies were performed by using the discharge tube as an integral part of the gas-sample system. This arrangement permits rapid gas sampling; 165 exploratory analyses were completed.

Preliminary experiments were designed and performed to provide a measure of resistor linearity as a function of the voltage applied to the input resistors of the vibrating reed electrometers which are used as detectors of positive ions on the mass spectrometers. Results for voltages from 0.5 to 8.0 volts on Victoreen 1 x 10^{10} ohm resistors indicate an appreciable change in resistance with applied voltage, of the order of 5 to 10%. The RC discharge time of the resistor through a known capacitor is used with a Keithly electrometer as an indicating device. Since most ion beams developed in the spectrometer generate less than 1 volt, this lower region will be further studied in some detail. (AEC Activity 5200)

Research and Development, Stable Isotopes - Approximately 370 grams of high purity elemental rubidium was prepared by a batch-type vacuum reduction-distillation procedure for use by ORSORT personnel in the determination of the physical properties of rubidium. Isotope targets and special fabrications prepared for nuclear investigations include (1) cylindrical slugs of stainless steel-clad Li^7, normal sodium, and normal potassium, (2) a 3-in.-dia. hollow Li^7 sphere, and (3) deposition of $Li_2^6SO_4$ in thin films on tantalum sheet stock by flame spraying. (AEC Activity 5200)

CHEMISTRY

Chemical Physics - The reaction between molecular beams of potassium and HBr was studied at a higher potassium temperature than before through the use of a new oven. One preliminary experiment with potassium at 840°K. and HBr at 460°K. was completed, and, taken with previous experiments with potassium at 541°K., indicates an activation energy of about 3.5 kcal. The distribution function for energies of collision of beams of potassium and of HBr over the range of temperatures of interest was computed on the Oracle.

Very intense atomic hydrogen hyperfine lines were examined by paramagnetic resonance in samples of H_2SO_4 and $HClO_4$ irradiated at 77°K with gamma rays. Each of the two hyperfine lines was found to have a pair of very weak satellite lines. Current experimental and theoretical work indicate that the satellite lines originate from environmental protons that are weakly coupled to the atomic hydrogen. The spacing of the satellite lines should be the proton nuclear magnetic resonance frequency in the particular field used. This was the case for nominal fields of 8,300 gauss, and equipment is being prepared for making similar tests at much lower fields. (AEC Activity 5300)

Chemistry of the Solid State - The determination of liquid-solid phase equilibria in cesium metal-cesium halide systems (excluding CsBr-Cs), which is all but completed, gives no indications for the co-existence of two liquid phases. Rather, the melting points of the salts are continuously lowered by increasing metal concentration. In other words, complete miscibility of the salts with the metal exists at the melting points of the salts. The similarity of heats of solution, at saturation, in the liquid metal for solid chloride and iodide is in agreement, as postulated, with the similarity of both the

heats of vaporization of these salts and of the energies assumed to be required for substitution of a Cl^- or an I^- ion for an electron of the liquid metal.

High-temperature x-ray diffraction measurements up to at least 3° below the melting point showed that CsBr, similarly to CsI, and differently from CsCl, does not undergo a solid phase transition from eight to six-fold coordination. The volume change on melting in both the bromide and iodide are, therefore, considered strong indication that this coordination change takes place on melting. (AEC Activity 5300)

Radio-Organic Chemistry - In the continuing investigation of the chemistry of phosphorus compounds, some of which are valuable in solvent extraction, two problems were pursued. Dehydration of orthophosphoric acid had been noted previously during experiments on the exchange of the butyl group between tributyl phosphate and labeled phosphoric acid. The dehydration was now studied as a function of temperature, by using selective elution from anion-exchange resins to separate the different products of dehydration. At 100°C, pyrophosphoric acid was the only dehydrated product, and the equilibrum mixture (observed to be the same at 20 and at 160 hrs) contained 4% of the phosphorus as this acid. At 175°C, the composition (based on phosphorus) was orthophosphoric acid, 49%; pyrophosphoric acid, 40%; triphosphoric acid, 9%; and tetraphosphoric acid, 2%. The composition was constant after 10 hrs.

A second problem was the preparation of pure di(2-ethylhexyl) hydrogen phosphate which is a useful solvent and which was needed for some fundamental studies. It is readily available in a mixture with the corresponding free alcohol and the tertiary ester as the surface-active agent P-28 (Carbide and Carbon Chemicals Corp.). The procedure worked out for the isolation comprises sorption from ethanol solution on an anion exchanger, elution with ethanol containing NaCl, evaporation, and acidification. (AEC Activity 5300)

Radiation Chemistry - Although, in the radiation-induced decomposition of water, the back reactions of H_2 with O_2 and with H_2O_2 at 25°C are fairly well understood, little is known about the radiation-induced reactions at high temperatures. The reaction of H_2 and O_2 is of especial importance in high temperature aqueous reactors. Hydrogen and oxygen dissolved in water react stoichiometrically at 200°C under the influence of Co^{60} gamma rays to give water with a yield of 4.5 molecules per 100 ev. Although on irradiation at low temperature, H_2O_2 builds up to a concentration of $\sim 10^{-3}$ M, the steady state concentration at 200°C is $< 10^{-6}$ M. An important reaction at 200°C appears to be $HO_2 + H_2O_2 \rightarrow H_2O + OH + O_2$. A mechanism based upon this and other probably reactions was developed to explain the observed yield at 200°C in terms of presently accepted yields of the free radicals produced in the radiation-induced decomposition of water.

The oxidation state of chromium in reactor solutions under irradiation may be an important factor in the corrosion of stainless steel. Chromate and chromic solutions with and without added UO_2SO_4 and $CuSO_4$ were irradiated with gamma rays under a variety of conditions. Chromate is reduced with a yield of 2.4 equivalents per 100 ev on irradiating oxygen-swept, 0.8 N sulfuric acid solutions with cobalt gamma rays at 25°C. With hydrogen sweeping, the yield is ~4.5. In oxygen-swept Cr^{+3} solutions in 0.8 N sulfuric acid, 2.2 equivalents of H_2O_2 are produced per 100 ev. In oxygen-swept, neutral chromate solutions, no reduction occurs on irradiation, and H_2O_2 is produced with a yield of 2.2 equivalents per 100 ev; with hydrogen sweeping, chromate is reduced with a yield of ~3.9 equivalents per 100 ev. At pH 3, gamma radiation produces no change at all in oxygen-saturated chromate solution, whereas with hydrogen sweeping, chromate is again reduced with a yield of ~4.8 equivalents. In oxygen-swept chromic solution at pH 3, chromate is produced with a yield of 1.2 equivalents per 100 ev. These results are consistent with results obtained in other solutions (e.g. ceric sulfate), and can be explained on the basis of proposed yields of free radicals in the radiation-induced decomposition of the water.

Although 10^{-3} M chromate in oxygen-saturated solution at pH 3 is not reduced by irradiation at 25°C, it is reduced in the presence of 0.12 M UO_2SO_4 and 0.02 M $CuSO_4$. By adding UO_2SO_4 or $CuSO_4$ separately, it was established that the reduction is promoted by UO_2SO_4. With all three solutes present, the chromate is not reduced at 250°C, but instead a yellow precipitate appears. This precipitate forms as readily without as during irradiation. The thermal oxidation of Cr^{+3} is rapid at this temperature and explains the absence of chromate reduction at 250°C.

On heating a hydrogen-saturated solution to 250°C, U_3O_8 precipitates and the chromate is reduced. Thermal processes at 250°C apparently mask most of the effects of gamma radiation in these systems. (AEC Activity 5300)

Chemistry of Fused Salts - The heat of combustion of boron carbide, B_4C, at 30 atm of oxygen was found to be 683.5 kcal/mole or 12.36 kcal/gram. The standard heat and free energy of formation of this substance were evaluated from the heat of combusion and other established thermal data giving $\Delta H_f^o{}_{298.16} = -14.1$ kcal/mole and $\Delta F_f^o{}_{298.16} = -13.9$ kcal/mole.

The heat capacity and related thermodynamic quantites were measured for molybdic oxide, MoO_3, in the range from 15° to 300°K. and it was found that the heat capacity varies as T^2 between 15° and 65°K. Molybdic oxide possesses layer-lattice structure and is the first example of a substance with more than two atoms per molecule which exhibits low-temperature T^2 heat capacity dependence. The measurements demonstrate that previous extrapolations below 70°K according to the Debye T^3 law are incorrect. At 25°C $C_p^o = 17.93$ cal/mole deg and $S^o = 18.58$ eu. It is planned to determine whether or not the heat capacity of molybdenum disulfide, MoS_2, which possesses a fairly prounounced layer-lattice, will obey a T^2 relation in the low temperature range.

Considerably improved techniques were developed for measuring the self-diffusion of ions in molten salts. The heat of activation for self-diffusion of Na^+ in molten $NaNO_3$ up to 645°C was estimated to be approximately 6 kcal/mole.

Detailed measurements of the molar volume and equivalent conductance of the molten salt system KCl-KI show its behavior to be consistent with predictions based on a previous study of similar systems. (AEC Activity 5300)

Uranium Chemistry of Raw Materials - Unsaturated straight chain secondary amines (e.g., Armeen 2S and Armeen 2T) have proved to be favorable reagents for the solvent extraction of uranium from sulfate liquors. These amines, available at a low cost, may be used in a kerosene diluent (possibly modified with a small amount of a long-chain alcohol), whereas the saturated straight chain secondary amines require more expensive aromatic diluents. Other amines wherein the alkyl groups are branched as well as unsaturated may be even more compatible with a kerosene diluent, as indicated from preliminary results with the Rohm and Haas compound EB-765-2.

As reported previously, loss of both long-chain amine reagent and hydrocarbon siluent was high when they were used to extract uranium from an acidified slurry of Florida Leached Zone ore. This loss apparently involved reaction with the solids and was more nearly proportional to the amount of slurry contacted than to the amine concentration. With very gentle agitation, the loss approached its maximum less rapidly than did the uranium extraction. In similar tests with mono- and di-alkylphosphoric acids the reagent loss to slurry was higher than to clarified liquor, but it was less than the corresponding loss of amine and involved less diluent loss. With all reagents, heavy emulsions were encountered unless the agitation (phase mixing) was very gentle.

Solubility measurements in the UO_2-P_2O_5-Cl_2O_7-H_2O system were made at 25°C in the range from 1.15 to 11.86 M $HClO_4$. The solid phases, regions of stability and solubilities are tentatively ascribed as follows:

	$[HClO_4]$	$[U^{+4}]$
$U(HPO_4)_2.6H_2O$	1.15 - 5.7	2.4×10^{-4} - 2×10^{-3}
$U(HPO_4)_2$ (metastable)	5.7 - 11.86	2×10^{-3} - 0.44
$U(HPO_4)_2,2H_2O$	5.7 - 7.0	2×10^{-3} - $.5\times10^{-4}$
$U(HPO_4)_2(HClO_4)_2.6H_2O$	7.0 - 11.25	5.5×10^{-4} - 2.5×10^{-2}
$U(HPO_4)_2(HClO_4)_2.4H_2O$	11.25 - 11.86	2.5×10^{-2} - 1.5×10^{-3}

(AEC Activity 5361)

Volatility Studies - In preliminary experiments the variables indicated to be the most important in controlling the rate of metal dissolution in fused fluoride baths, for eventual processing by a volatility method, were bath composition, temperature, hydrogen fluoride feed rate, and metallurgical history of the metal. By selection of a suitable combination of bath composition and temperature, the penetration rate for uranium could be varied from 0.1 to 17 mils/hr and for zirconium from 22 to 288 mils/hr. The independent effect of temperature on the zirconium dissolution rate was rather slight in a typical bath (ZrF_4-KF-NaF = 44.5-48.5-7 mole %), but the rate doubled for every 140° rise in temperature in the vicinity of 600°C. With zirconium, changing the hydrogen fluoride feed rate from 83 to 280 millimoles/hr approximately doubled the penetration rate at 675°C. This effect was probably primarily due to greater agitation of the salt with the more rapid gas flow and was of approximately the same magnitude as in other types of diffusion-limited surface reactions. A zirconium specimen that had undergone considerably less cold rolling in preparation than a second specimen was found to be less reactive by a factor of 2.5.

Preliminary data indicated that the boundary between laminar and turbulent flow of fluorine through molten salt is marked by a sharp break in the plot of friction factor vs the Reynolds number. It is hoped that determination of the pressure drop at which the break occurs will indicate the gas throughput required to produce turbulence and therefore efficient contact of gas and melt. (AEC Activity 5300)

Thorex Process Development - Aluminum-silicon bonded thorium slugs were more difficult to process than nonbonded slugs; they were extremely resistant to conventional acidic or caustic dissolution methods. Inter-metallic compounds of thorium silicide had formed on the interface between the thorium and the aluminum-silicon, resulting in a brittle, highly corrosion resistant, fractured layer about 7 mils thick.

Laboratory-scale experiments indicated that the present Thorex flowsheet, which includes a feed evaporation-digestion step, can be used to process FMPC zirconia-cast thorium metal as well as the previously tested berylliacast thorium. The zirconia-cast thorium contains more silica than berylliacast metal.

In tests of the present Thorex flowsheet, with both feed and scrub acid-deficient, less mercury and fluoride appeared to be extracted than in the previous one using acid feed. Mercury and fluoride concentrations of less than 25 and about 700 ppm of thorium, respectively, can be expected in the thorium product.

Construction of the Thorex pilot plant was 96% completed except for the unit shielding which is to be installed prior to operation with radioactive material. The thorium nitrate tetrahydrate digester, for preparation of feed for the initial, nonradioactive runs, was put in operation. (AEC Activity 5300)

Analytical Research - Since UF_3 has been shown to exhibit a marked inhibitory effect on corrosion by fused fluoride reactor fuels, considerable effort is being devoted to studies of methods for analyzing UF_3 - UF_4 mixtures in fused salt matrices. In an effort to analyze for UF_4, studies were made of the polarography of uranium in molten ammonium formate. Quite contrary to work reported in the literature, it was found that U(IV) did not give a reduction wave in molten ammonium formate. It was found that aerated solutions of U(IV) or solutions of U(VI) in ammonium formate do give reduction waves with half wave potentials of about -0.2 volt against a mercury pool anode. It is hoped that this type of polarography will prove analytically useful even though the reported reduction wave for U(IV) does not seem to exist.

Coulometric titration methods are being studied for possible application to remote analytical procedures on radioactive samples. The determination of chloride by coulometric titration was successful over a range from 1 to 100 micrograms. It was also shown to be feasible to determine iron in the presence of uranium by coulometric titration by using electrolytically generated U(V or IV) as the reductant. It is hoped that the latter titration will also prove feasible for the determination of Pu in the presence of large amounts of uranium, but the precision obtained in this case has not been fully satisfactory. Both of these titrations have possible applications to HRT samples.

METALLURGY

Ceramics Research - Investigation of clays in connection with a proposal for sealing projected waste pits indicated that Tennessee ball clay and also a mixture of 25% bentonite and 75% Conasauga shale showed promise of providing an impervious layer in contact with waste solution. Studies on the disposal of high level radioactive wastes suggest that a gel prepared by reacting limestone with the Hope solution will provide a method for retaining much of the Sr^{90} contained in the solution. The nitrates present, however, are not held by the gel. In the course of development of a ceramic-to-metal seal for special applications at K-25, it was found that specimens of dense alumina sprayed with copper and also specimens coated with silver paste could be soldered with a lead-silver solder. (AEC Activity 5400)

Physical Metallurgy of Reactor Materials - In continuation of the study of carburization of stainless-steel fuel elements to improve chemical dissolution it was found that 2% or more carbon is desirable. This amount of carbon can be obtained with a carburization treatment lasting for not more than one hour, but this treatment must be followed by holding the fuel elements at an elevated temperature in order that the carbon may diffuse into the interior of the element. Treatment of full-size fuel elements is now being considered.

Impact properties of thorium were determined over a temperature range from -196°C to 200°C. Thorium made by the iodide process behaves as do other face-centered cubic metals in that impact strength increases with decreasing temperature, and it shows no transition in the impact resistance-

temperature curve. The addition of as little as 0.05% carbon to "iodide" thorium results in a transition in the impact resistance-temperature curve, the impace resistance decreasing rapidly with temperature at the so-called transition temperature. (AEC Activity 5400)

Fundamental Physico-Metallurgical Research - Recent examination of Zr-Sb alloys of increased purity confirms the phase relationships found earlier in the region of the peritectoid reaction $\beta + Zr_2Sb \longrightarrow \alpha$, except that the reaction temperature was found to be 10°C higher; the present value is 880°C.

Calculations of the α/β transformation boundaries in titanium and zirconium alloy systems were extended to include the case where the density of states of the α phase is assumed to increase with decreasing electron concentration below two electrons/atom. These calculations give better agreement in some systems, such as Zr-Ag. The density of states variation in hexagonal Zr-Ag alloys will be determined by low temperature specific heat measurements to confirm the validity of this assumption for these alloys.

A thorough examination was made of the preferred orientation of some cold-rolled aluminum sheet. The texture was treated as three fiber textures about mutually perpendicular axes. Pole distribution and axis distribution charts were obtained, taking as "fiber axes" the rolling, normal, and transverse directions. For comparison, a conventional pole figure was obtained. The former method was found to give a much more precise indication of the nature of the preferred orientation and more directly the information desired. The principal component of the texture was near (513)[334]. The result is at variance with that reported by previous investigators, who have found (123) [121] to be the principal component. (AEC Activity 5400)

Fundamental Investigations of Radiation Damage - The magnetic susceptibility vs temperature curve of a second irradiated specimen of piezo-crystal quartz was measured. This bombardment of 3.3×10^{20} fast neutrons/cm^2 at 100°C, the longest irradiation of any magnetically studied quartz crystal, apparently produced only 2.5×10^{19} paramagnetic centers of spin 1/2 as compared to 6.2×10^{19} after a bombardment of 5×10^{19} neutrons/cm^2 as previously reported. The long irradiation resulted in a greater density decrease (14% as compared to 2%). The crystal had a faint magenta coloration. Curie plots of the difference data (unirradiated subtracted from irradiated) indicate values of a Wiess constant which increase with irradiation. A least squares analysis of the primary data using $-\chi \times 10^7 = \frac{A}{T+\Delta} + B + CT$ as a trial form of the equation does not yield conclusive values of the constants. (AEC Activity 5411)

BIOPHYSICS

Waste Disposal Research - Several runs were completed with the process waste treatment experimental plant facilities for investigating processes for reducing the amount of radioactive materials discharging from the ORNL Settling Basin. The removals obtained by using upflow solids contact-type units with either alum or ferric chloride coagulation, phosphate coagulation, or excess lime soda ash softening have not been entirely adequate to meet the levels arbitrarily selected (90% removal of activity.) Additional studies are underway to evaluate the efficacy and economics of ion exchange processes, either with or without previous chemical treatment of the wastes, for meeting this criterion.

As part of the liquid waste disposal studies, a field permeability test utilized by the Bureau of Reclamation was employed to determine the coefficients of permeability of the weathered Conasauga shale. It is referred to as a well permeameter test, and consists of rate of flow measurements from an uncased well in which a constant head of water is maintained. Permeability values ranging from 120 ft/year to 710 ft/year were obtained from five test locations in the four-acre test area (ORNL 1748, p. 25).

A study to determine the uptake of radioactive isotopes by treated clays was undertaken in cooperation with the group of 21 AEC Fellows in Radiological Physics. Samples of a local clay were saturated with each of several cations (H^+, Cs^+, Sr^{++}, Y^{+++}, Ru^{+++}, and Zr^{4+}). These treated aggregates were shaken with various radioactive solutions containing a single radioisotope (Cs^{137}, Sr^{90}, Y^{90}, Ru^{106}, and Zr^{95}-Nb^{95}) and the uptake of each of these isotopes by the treated clays was determined. With the exception of Cs, the adsorption of the radioisotopes by the specific soil under test at pH of 3.0 to 5.0 was in accordance with the order predicted by the lyotropic series. At higher H^+ ion concentrations, the removal percentages decreased. (AEC Activity 6510)

Radioactive Particulate Problem - The effectiveness of ordinary dry river sand (median diameter 0.007 inches) in filtering aerosols was determined by using a 5-in. thick sand column. At air flow rates verying from 0.04 to 10 ft/min, the aerosol size for maximum penetration was found to be between 0.2 and 0.3 micron radius, and the aerosol penetration, for this size, ranged from about 0.1% at 0.04 ft/min to about 15% at 10 ft/min.

At the "boil-up" rate of 0.04 ft/min calculated (ORNL-1638) for the "Project Hope" waste disposal pit less than 3 ft of sand will be adequate to provide an overall decontamination factor of 10^{12}. This extrapolation from laboratory results assumed (1) that a decontamination factor of about 10^5 - 10^7 will be obtained by boiling alone (ORNL-1748, p. 26), and (2) the sand is dry. (AEC Activity 6520)

Professional Health Physics Training - The 21 AEC Fellows in Radiological Physics who started their training in Applied Health Physics at the Laboratory on June 8, completed the training August 27. Of the nine who accepted extensions of their Fellowship, seven will return to Vanderbilt and two will remain at the Laboratory to pursue a research problem in Health Physics to fulfill the thesis requirement for a master's degree. Nine have accepted positions in Health Physics, one in the teaching of science and mathematics, and two are returning to universities for further graduate work. (AEC Activity 7620)

RADIOISOTOPE SALES AND COSTS

Type of Transaction	July	FY to Date
Domestic Sales	$ 79,679	$ 79,679
Foreign Sales	6,934	6,934
Project-Cash Sales	7,215	7,215
Project-Transfer Credits	305	305
Technical Cooperation Program Credits	-0-	-0-
Plant Credits	4,042	4,042
Civilian Defense Credits	-0-	-0-
Cancer Program Credits	18,629	18,629
*Miscellaneous Income	23	23
Total Income	$116,827	$116,827
**Income-No Production costs incurred Subtract for Total Income	-149	-149
Net Radioisotope Income	$116,976	$116,976
Radioisotope Costs	$ 66,418	$ 66,418
Radioisotope Shipments	977	977

* Miscellaneous Income - D_2O, Freight, Missing Parts, TCP.
**Income from H^3, He^3, B^{10}, and miscellaneous items in excess of handling costs.

GROSS OPERATING COSTS

	Cost for July	FY 1955 Cost to Date
Programmatic Operating Cost - Net	$ 2,669,715	$ 2,669,715
Plant and Equipment Cost	174,749	174,749
Construction Program "H"	5,643	5,643
Work for Other Parties - Transfers	22,798	22,798
Inventory Changes	31,686*	31,686*
Reimbursable Work for Other Parties	138,064	138,064
Deferred Charges	172*	172*
Total Laboratory Cost - Net	$ 2,979,111	$ 2,979,111
Estimated Cost for Next Month - Net	$ 3,000,000	$ 5,979,111

*Credit

[redacted]

PERSONNEL SUMMARY

	Number of Employees August, 1954	New Hires August	Terminations August
Administration	61	0	3
Operations*	122	0	0
Engineering, Shops and Mechanical	817	4	15
Laboratory and Research	1902	43	68
Protection	147	1	0
Service	380	3	6
	3429	51	92

*Includes Electrical Distribution and Steam Plant as well as the Operations Division.

A total of 744 Laboratory personnel are located in the Y-12 Area.

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March 1954	ORNL-1703
April 1954	ORNL-1719
May 1954	ORNL-1733
June 1954	ORNL-1748
July 1954	ORNL-1760

[redacted]